

Organic & Biomolecular Chemistry

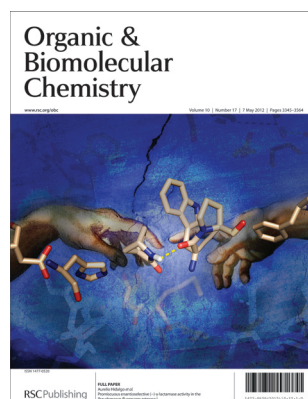
An international journal of synthetic, physical and biomolecular organic chemistry

www.rsc.org/obc

RSC Publishing is a not-for-profit publisher and a division of the Royal Society of Chemistry. Any surplus made is used to support charitable activities aimed at advancing the chemical sciences. Full details are available from www.rsc.org

IN THIS ISSUE

ISSN 1477-0520 CODEN OBCRAK 10(17) 3345–3564 (2012)



Cover

See Aurelio Hidalgo *et al.*, pp. 3388–3392.

Cover art by J. Belio.

Image reproduced by permission of Aurelio Hidalgo from *Org. Biomol. Chem.*, 2012, **10**, 3388.

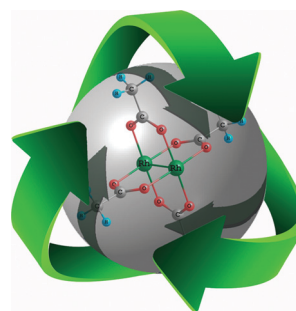
PERSPECTIVE

3357

Making expensive dirhodium(II) catalysts cheaper: Rh(II) recycling methods

Nuno R. Candeias,* Carlos A. M. Afonso and Pedro M. P. Gois*

A combined overview of the available methods for recovering and reusing dirhodium(II) metal complexes in catalysis, covering homogeneous catalysis as well as heterogenisation methods, is documented.



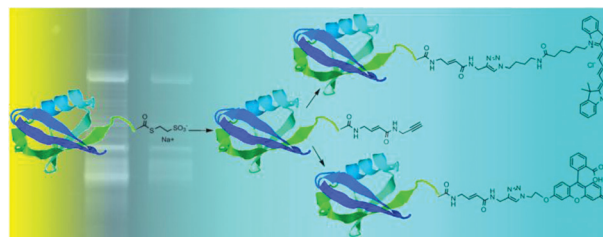
COMMUNICATIONS

3379

Fluorescence-based active site probes for profiling deubiquitinating enzymes

Joanna F. McGouran, Holger B. Kramer, Mukram M. Mackeen, Katalin di Gleria, Mikael Altun and Benedikt M. Kessler*

Novel fluorescent ubiquitin-based active site probes allow detection of endogenous deubiquitinating enzyme activities in cell extracts by in-gel fluorescence imaging.



EDITORIAL STAFF

Editor

Richard Kelly

Deputy editor

Marie Cote

Development editor

Francesca Burgoyne

Senior publishing editor

Helen Saxton

Publishing editorsNicola Burton, Sarah Dixon, Scott Gallifent-Holmes,
Frances Galvin, Elisa Meschini, Roxane Owen**Publishing assistants**

Aliya Anwar, Nathalie Horner

Publisher

Emma Wilson

For queries about submitted papers, please contact Helen Saxton, Senior publishing editor in the first instance. E-mail: obc@rsc.org

For pre-submission queries please contact Richard Kelly, Editor. Email: obc-rsc@rsc.org

Organic & Biomolecular Chemistry (print: ISSN 1477-0520; electronic: ISSN 1477-0539) is published 24 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF.

All orders, with cheques made payable to the Royal Society of Chemistry, should be sent to RSC Distribution Services, c/o Portland Customer Services, Commerce Way, Colchester, Essex, UK CO2 8HP.

Tel +44 (0)1206 226050; E-mail sales@rscdistribution.org

2011 Annual (print+electronic) subscription price: £3726; US\$6955. 2011 Annual (electronic) subscription price: £3353; US\$6260. Customers in Canada will be subject to a surcharge to cover GST. Customers in the EU subscribing to the electronic version only will be charged VAT. If you take an institutional subscription to any RSC journal you are entitled to free, site-wide web access to that journal. You can arrange access via Internet Protocol (IP) address at www.rsc.org/ip. Customers should make payments by cheque in sterling payable on a UK clearing bank or in US dollars payable on a US clearing bank.

Periodicals postage paid at Jamaica NY 11431.

US Postmaster: Send address changes to Organic & Biomolecular Chemistry, Air Business Ltd, c/o Worldnet Shipping Inc., 156-15, 146th Avenue, 2nd Floor, Jamaica, NY 11434, USA.

The Royal Society of Chemistry takes reasonable care in the preparation of this publication but does not accept liability for the consequences of any errors or omissions. Inclusion of an item in this publication does not imply endorsement by The Royal Society of Chemistry of the content of the original documents to which that item refers.

Advertisement sales: Tel +44 (0) 1223 432246;
Fax +44 (0) 1223 426017; E-mail advertising@rsc.org

For marketing opportunities relating to this journal, contact marketing@rsc.org

Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

www.rsc.org/obc

Organic & Biomolecular Chemistry brings together molecular design, synthesis, structure, function and reactivity in one journal. It publishes fundamental work on synthetic, physical and biomolecular organic chemistry as well as all organic aspects of: chemical biology, medicinal chemistry, natural product chemistry, supramolecular chemistry, macromolecular chemistry, theoretical chemistry, and catalysis.

EDITORIAL BOARD

Chair

Jeffrey Bode, ETH Zürich, Switzerland

Associate Editors

Jin-Quan Yu, Scripps Research

Institute, La Jolla, CA, USA

Andrei Yudin, University of Toronto,

Canada

Ashraf Brik, Ben-Gurion University of
the Negev, Israel

Margaret Brimble, University of

Auckland, New Zealand

Pauline Chiu, University of Hong

Kong, China

Anthony Davis, University of Bristol,
UKVeronique Gouverneur, University of
Oxford, UKChristian Hertweck, Leibniz-Institute
Jena, Germany

Kenichiro Itami, Nagoya University,

Japan

Stephen Kent, University of Chicago,

USA

Paolo Scrimin, University of Padova,
Italy

Qi-Lin Zhou, Nankai University, China

ADVISORY BOARD

Helen Blackwell, University of
Wisconsin-Madison, USA

Barry Carpenter, Cardiff University, UK

Michael Crimmins, University of North
Carolina, USAAntonio Echavarren, Autonomous
University of Madrid, Spain

Jonathan Ellman, Yale University, USA

Kurt Faber, University of Graz, Austria

Ben Feringa, University of Groningen,
Netherlands

Nobutaki Fujii, Kyoto, Japan

Jan Kihlberg, Umeå University, Sweden

Philip Kocienski, University of Leeds,
UKSteven V Ley, University of Cambridge,
UKStephen Loeb, University of Windsor,
CanadaIlan Marek, Israel Institute of
Technology, Israel

Manuel Martín Lomas, CCRB, San

Sebastián, Spain

Keiji Maruoka, Kyoto University, Japan

Heather Maynard, University of

California, Los Angeles, USA

E W 'Bert' Meijer, Eindhoven University
of Technology, NetherlandsEiichi Nakamura, University of Tokyo,
Japan

Ryoji Noyori, Nagoya University, Japan

Mark Rizzacasa, University of

Melbourne, Australia

Richmond Sarpong, University of

California, Berkeley, USA

Oliver Seitz, Humboldt University of
Berlin, Germany

Bruce Turnbull, University of Leeds, UK

Chris Welch, Merck & Co., Rahway,
NJ, USAHelma Wennemers, University of Basel,
SwitzerlandPeter Wipf, University of Pittsburgh,
USAHenry N C Wong, Chinese University of
Hong Kong, ChinaShuli You, Shanghai Institute of
Organic Chemistry, China

Sam Zard, Ecole Polytechnique, France

Zhang Li-He, Beijing Medical
University, China

INFORMATION FOR AUTHORS

Full details on how to submit material for publication in *Organic & Biomolecular Chemistry* are given in the Instructions for Authors (available from <http://www.rsc.org/authors>). Submissions should be made *via* the journal's homepage: <http://www.rsc.org/obc>.

Authors may reproduce/republish portions of their published contribution without seeking permission from the RSC, provided that any such republication is accompanied by an acknowledgement in the form: (Original Citation)—Reproduced by permission of The Royal Society of Chemistry.

This journal is © The Royal Society of Chemistry 2012. Apart from fair dealing for the purposes of research or private study for non-commercial purposes, or criticism or review, as permitted under the Copyright, Designs and Patents Act 1988 and the Copyright and Related Rights Regulation 2003, this publication may only be reproduced, stored or transmitted, in any form or by any means, with the prior permission in writing of the Publishers or in the case of reprographic reproduction in accordance with the terms of licences issued by the Copyright Licensing Agency in the UK. US copyright law is applicable to users in the USA.

The Royal Society of Chemistry takes reasonable care in the preparation of this publication but does not accept liability for the consequences of any errors or omissions.

Ⓢ The paper used in this publication meets the requirements of ANSI/NISO Z39.48–1992 (Permanence of Paper).

Royal Society of Chemistry: Registered Charity No. 207890.

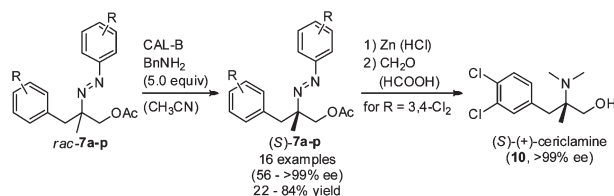
COMMUNICATIONS

3384

Synthesis of (*S*)-(+)-cericlamine through lipase-catalyzed aminolysis of azo acetates

Agnes Prechter, Harald Gröger and Markus R. Heinrich*

The kinetic enzymatic resolution of azo acetates *via* aminolysis with *Candida antarctica* lipase B has been investigated. The products obtained from this biotransformation can serve as valuable precursors for various amino alcohols, as exemplified by the synthesis of the serotonin reuptake inhibitor (*S*)-(+)-cericlamine.



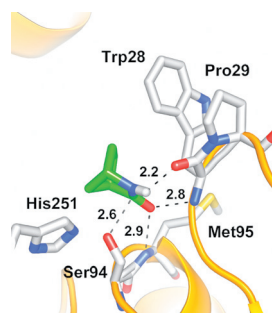
PAPERS

3388

Promiscuous enantioselective (–)- γ -lactamase activity in the *Pseudomonas fluorescens* esterase I

Leticia L. Torres, Anna Schließmann, Marlen Schmidt, Noella Silva-Martin, Juan A. Hermoso, José Berenguer, Uwe T. Bornscheuer and Aurelio Hidalgo*

A promiscuous but very enantioselective (–)- γ -lactamase activity in the kinetic resolution of the Vince lactam (2-azabicyclo[2.2.1]hept-5-en-3-one) was detected in the *Pseudomonas fluorescens* esterase I (PFEI).

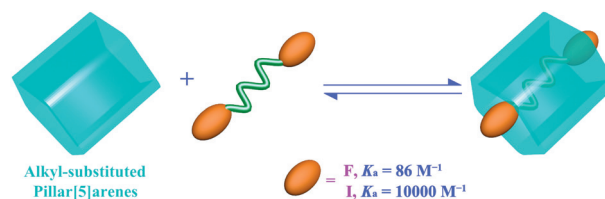


3393

Complexation of neutral 1,4-dihalobutanes with simple pillar[5]arenes that is dominated by dispersion forces

Xiaoyan Shu, Jiazeng Fan, Jian Li, Xiaoyang Wang, Wei Chen, Xueshun Jia* and Chunju Li*

The complexation of neutral 1,4-dihalobutanes with simple pillar[5]arenes was investigated, indicating the formation of interpenetrated complexes, where the dispersive interactions dominate the complex stability.

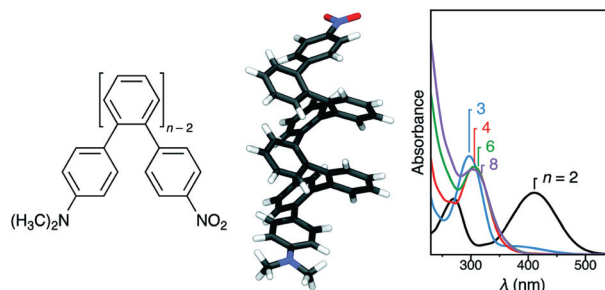


3398

***ortho*-Phenylene oligomers with terminal push–pull substitution**

Jian He, Sanyo M. Mathew, Sarah D. Cornett, Stephan C. Grundy and C. Scott Hartley*

Substituent effects on conformational behavior and electronic spectra have been examined in the first series of push–pull *o*-phenylenes.



Click through to a great career

@ ChemCareers 2012

THE CAREERS FAIR WITH A DIFFERENCE



29 - 30 May 2012

Hosted on MyRSC, the online community for the chemical sciences

Explore what options are available in chemistry by taking part in this exciting online event.

ChemCareers will allow you to:

- **Discover** the huge range of career opportunities available to chemical scientists
- **Learn** how to market yourself to employers in job applications and interviews
- **Seek** expert advice on career planning and making your next career move
- **Investigate** further study options

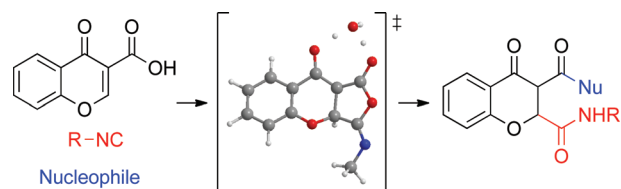
Register now at <http://my.rsc.org/chemcareers> and sign up to the ChemCareers group to keep up-to-date with the full programme of events planned for 2012.

3406

Conjugate addition of isocyanides to chromone 3-carboxylic acid: an efficient one-pot synthesis of chroman-4-one 2-carboxamides

Ana G. Neo, Jesús Díaz, Stefano Marcaccini and Carlos F. Marcos*

Isocyanides act as nucleophiles in the Michael addition to chromones.

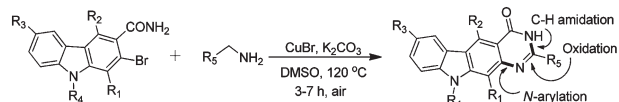


3417

Copper-mediated domino synthesis of pyrimido[4,5-*b*]carbazolones *via* Ullmann *N*-arylation and aerobic oxidative C–H amidation

Devanga K. Sreenivas, Nagarajan Ramkumar and Rajagopal Nagarajan*

New pyrimido[4,5-*b*]carbazolone derivatives have been synthesized through cascade Ullmann *N*-arylation and aerobic oxidative C–H amidation reactions catalyzed by CuBr under air and ligand-free conditions.

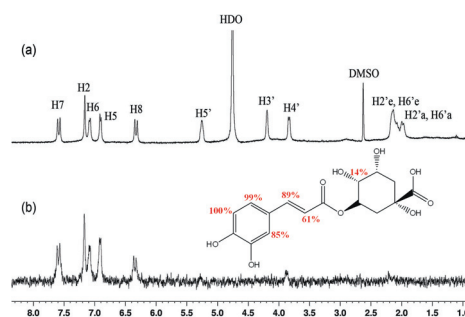


3424

Characterization of hydroxycinnamic acid derivatives binding to bovine serum albumin

Xiao-Ling Jin, Xia Wei, Feng-Ming Qi, Sha-Sha Yu, Bo Zhou* and Shi Bai*

This study provides the detailed information for elucidating the interaction of bovine serum albumin and hydroxycinnamic acid derivatives by NMR spectroscopic techniques in combination with fluorescence and molecular modeling methods.

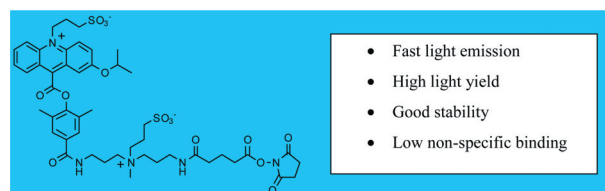


3432

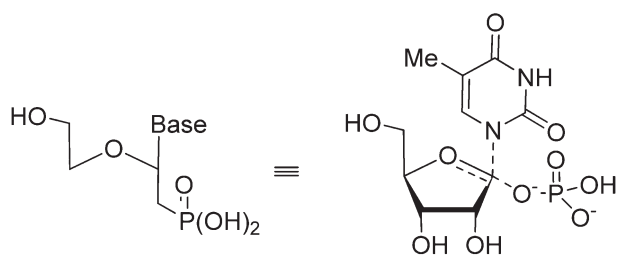
Chemiluminescence from alkoxy-substituted acridinium dimethylphenyl ester labels

Anand Natrajan,* David Sharpe and David Wen

Chemiluminescent acridinium esters with relatively hydrophobic acridinium rings and hydrophilic leaving groups display fast light emission and increased light output for applications in automated immunoassays.



3448

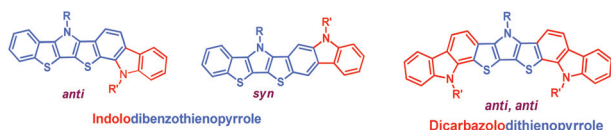


Synthesis of a new family of acyclic nucleoside phosphonates, analogues of TPases transition states

B n dicte Dayde, Samira Benzaria, Claire Pierra, Gilles Gosselin, Dominique Surleraux, Jean-No l Volle, Jean-Luc Pirat and David Virieux*

A 6-step procedure was developed for the synthesis of a new family of acyclic nucleoside phosphonates (ANPs), "PHEEPA" [(2-pyrimidinyl-2-(2-hydroxyethoxy)ethyl)phosphonic acids] in overall yields ranging from 4.5% to 32%.

3455

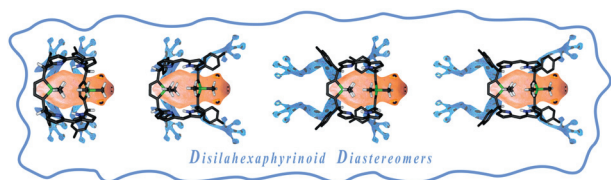


Synthesis and properties of thienopyrrole based heteroacenes – indolodibenzothienopyrrole and dicarbazolodithienopyrrole

Ganapathy Balaji, Andrea M. Della Pelle, Bhooshan C. Popere, A. Chandrasekaran and S. Thayumanavan*

Synthesis and characterization of stable and soluble heteroarenes up to nine fused rings.

3463

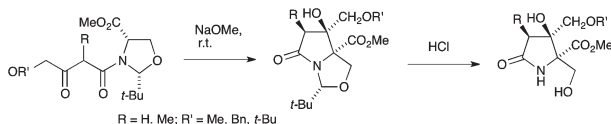


Core-modified hexaphyrin: synthesis and characterization of 31,34-disilahexaphyrinoid

Janusz Skonieczny, Lechosław Latos-Grażyński* and Ludmiła Szterenber

The condensation of 16-silatripyrrane with pentafluorobenzaldehyde affords only one isolable meso diastereomer of 31,34-disilahexaphyrinoid, the first which contains two built-in silole units.

3472



Biomimetic synthesis, antibacterial activity and structure–activity properties of the pyroglutamate core of oxazolomycin

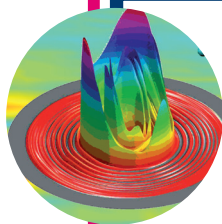
Plamen Angelov, Yui Kwan Sonia Chau, Paul J. Fryer, Mark G. Moloney,* Amber L. Thompson and Paul C. Trippier

Biomimetic intramolecular aldol reactions on oxazolidine templates derived from serine may be used to generate densely functionalised pyroglutamates, which are simpler mimics of the right hand side of oxazolomycin.

Faraday Discussions

June - September 2012

Register early for the best prices

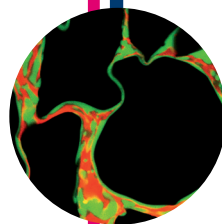


Molecular Reaction Dynamics in Gases, Liquids and Interfaces

Faraday Discussion FD157

25 - 27 June 2012 | Assisi, Italy

www.rsc.org/FD157



Soft Matter Approaches to Structured Foods

Faraday Discussion FD158

2 - 4 July 2012 | Hof van Wageningen, The Netherlands

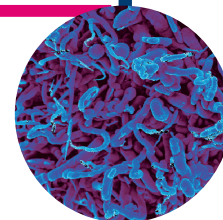
www.rsc.org/FD158

Crystallisation - A Biological Perspective

Faraday Discussion FD159

23 - 25 July 2012 | Leeds Metropolitan University, UK

www.rsc.org/FD159

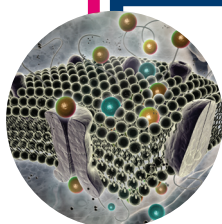
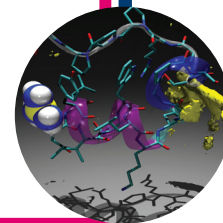


Ion Specific Hofmeister Effects

Faraday Discussion FD160

3 - 5 September 2012 | Queen's College Oxford, UK

www.rsc.org/FD160



Lipids and Membrane Biophysics

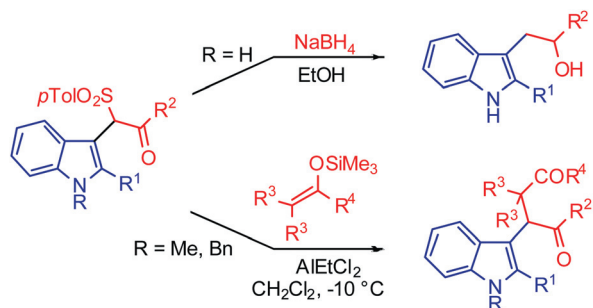
Faraday Discussion FD161

11 - 13 September 2012 | Burlington House, London, UK

www.rsc.org/FD161

For full details see individual event websites

3486

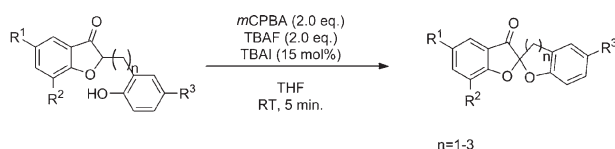


Ketosulfonyl indoles in the regiodefined synthesis of tryptophols and related indole derivatives

Alessandro Palmieri and Marino Petrini*

Ketosulfonyl indoles react with NaBH₄ leading to tryptophols in a regio-complementary fashion with respect to the traditional oxirane ring-opening by indoles. Compared to traditional β-ketosulfones, ketosulfonyl indoles show a peculiar behavior since they undergo a Lewis acid promoted elimination of the arylsulfonyl group allowing the preparation of indolyl-substituted 1,4-dicarbonyl derivatives.

3494

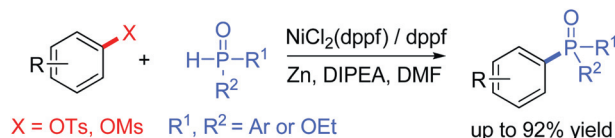


New fluoride-promoted hypiodite-catalytic oxidative cycloetherification to aromatic spiroketals

Wei Wei, Liqi Li, Xiaohong Lin, Haifeng Li, Jijun Xue* and Ying Li*

A new catalytic application of hypiodite reagents generated *in situ* from iodide ions is found, which succeeded in the synthesis of bisbenzannelated spiroketal cores for the first time. Fluoride was proven to be obligatory for this spiroketalization, which is the first fluoride-promoted oxidative cycloetherification to aromatic spiroketals.

3500

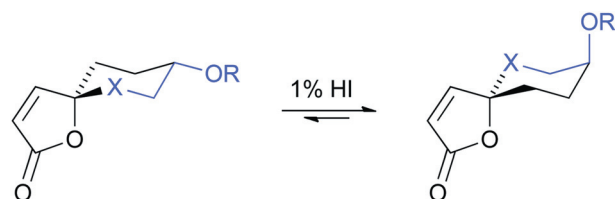


Nickel-catalyzed C–P coupling of aryl mesylates and tosylates with H(O)PR¹R²

Chaoren Shen, Guoqiang Yang and Wanbin Zhang*

The first method for the nickel-catalyzed phosphonylation of aryl mesylates and tosylates was developed with moderate to good yields.

3506



Conformational preferences of oxy-substituents in butenolide–tetrahydropyran spiroacetals and butenolide–piperidine spiro-*N,O*-acetals

Sébastien Naud, Sarah J. Macnaughton, Bryony S. Dyson, Daniel J. Woollaston, Jonathan W. P. Dallimore and Jeremy Robertson*

3-Oxy-substituents in butenolide spiroacetals (X = O) and spiro-*N,O*-acetals (X = NTs) show a modest axial preference in solution.

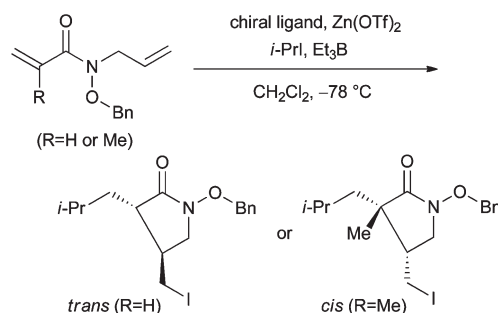
PAPERS

3519

Lewis acid-mediated radical cyclization: stereocontrol in cascade radical addition–cyclization–trapping reactions

Hideto Miyabe,* Ryuta Asada and Yoshiji Takemoto*

An efficient approach for achieving radical cyclizations by using hydroxamate as a coordination tether with Lewis acid was studied.

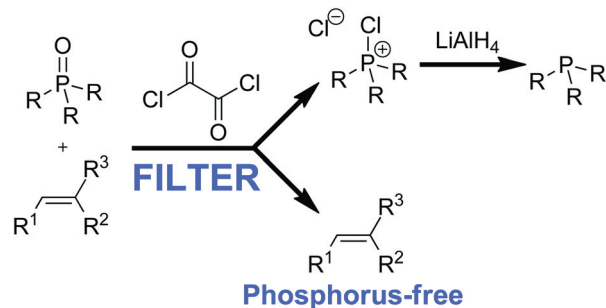


3531

A convenient and mild chromatography-free method for the purification of the products of Wittig and Appel reactions

Peter A. Byrne, Kamalraj V. Rajendran, Jimmy Muldoon and Declan G. Gilheany*

The product of any Wittig or Appel reaction is obtained phosphorus-free by means of a simple filtration.

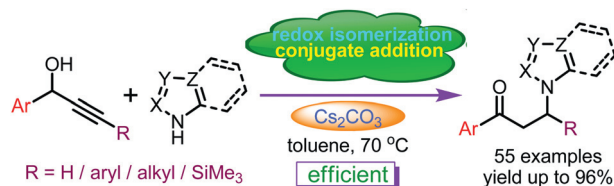


3538

A convenient approach to β -heteroarylated (C–N bond) ketones from Cs_2CO_3 promoted reaction between propargyl alcohols and nitrogen-heterocycles

M. Bhanuchandra, Malleswara Rao Kuram and Akhila K. Sahoo*

An efficient one-step approach to β -heteroarylated (C–N bond) ketones through base induced redox-isomerization conjugate addition of NH-heteroarenes with propargyl alcohols is demonstrated.

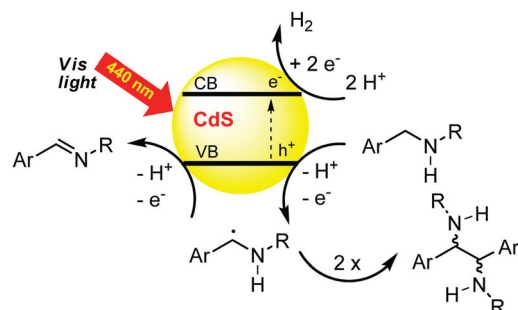


3556

Visible light mediated homo- and heterocoupling of benzyl alcohols and benzyl amines on polycrystalline cadmium sulfide

Tatiana Mitkina, Christoph Stanglmair, Wolfgang Setzer, Michael Gruber, Horst Kisch and Burkhard König*

The oxidative coupling of sp^3 hybridized carbon atoms by photocatalysis is a valuable synthetic method as stoichiometric oxidation reagents can be avoided and dihydrogen is the only byproduct of the reaction.



chemistryworldjobs

The best recruitment site dedicated to chemistry and the chemical sciences

Create a **free** account to get the most from chemistryworldjobs.com

● **Get headhunted**

Create a profile and publish your CV so potential employers can discover you

● **Stay ahead of your competition**

Set up your job alerts and receive relevant jobs in your inbox as soon as they appear

● **Discover your next career move**

Detailed searches by role, salary and location

● **Save time and be the first to apply**

Online vacancy application

● **Be efficient**

Bookmark jobs that interest you, so you can come back to them later

Upload your CV and be entered into a monthly draw for a **free CV review**

BE DISCOVERED...

